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ORIGINAL ARTICLES

Role of surface tension and roughness on the wettability of Er:YAG laser irradiated dentin: *In vitro* study

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Introduction: The aim of this "in vitro" study was to evaluate the role of surface tension and surface roughness in the wettability, considered essential for a good adhesion, comparing Er:YAG laser - to bur-prepared dentin.
Materials and Methods: Dentin surfaces of third human molars were Er:YAG laser- and bur-prepared to evaluate the effects of surface tension and roughness on wettability and interferometric analysis was used to compare the roughness of the two groups surfaces, after gold-coating them.
Results: In bur-prepared samples the time taken for the water drop to spread out was approximately the same with or without metallization while, in the Er:YAG laser-prepared surfaces the spreading-out time was less than 10 seconds but longer after metallization i.e. nearly two minutes. Large differences in wettability measurements were observed because the water drop was almost immediately absorbed on the Er:YAG laser-prepared surface. The wettability test demonstrated that the porous and hydrophilic properties of Er:YAG laser-prepared surfaces are higher than bur-prepared surfaces.
Conclusion: Surface tension, surface morphology and porosity had different effects on the spreading time of a water drop on both Er:YAG laser- and bur-prepared surfaces. And, while surface tension does not seem to influence the results, roughness appears to be the main parameter involved in water drop spreading, this being an indication, by the clinical point of view, to the choice of Er:YAG laser parameters in conservative dentistry.

Key words: Dentin・Er:YAG laser・roughness・wettability

Introduction

In 1990 laser technology was introduced in conservative dentistry by Hibst and Keller, who described the possibility to use an Er:YAG laser as alternative to conventional instruments, such as the turbine and micromotor [1,2]. Widespread interest in employing this new technology stems from a number of significant advantages, as described in several scientific studies. Thanks to the affinity of the Er:YAG laser wavelength (2940 nm) to water (absorption peak = 3000nm) and hydroxyapatite (absorption peak = 2800nm), laser technology allows for efficient ablation of hard dental tissues without the risk of micro- and macro-fractures, as have been observed with the use of conventional rotating instruments [3,4]. The dentin surface treated by laser appears clean, without a smear-layer and with the tubules open and clear [5].

Thermal elevation in the pulp, recorded during Er:YAG laser irradiation, is lower than that recorded by using a turbine and micro-motor with the same conditions of air/water spray [6,7]. This wavelength also has an antimicrobial decontamination effect on the treated tissue, which destroys both aerobic and anaerobic bacteria [8]. The most interesting aspects of this new tech-

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nology are related to the goals of modern conservative dentistry: i.e. minimally invasive treatments and adhesive dentistry. Er:YAG lasers can reach spot dimensions smaller than 1 mm, which enables a selective ablation of the affected dentin while preserving the surrounding sound tissue to produce highly efficient restorations [9]. Several in vitro studies have demonstrated that the preparation of enamel and dentine by Er:YAG laser, followed by orthophosphoric acid-etching, enhances effectiveness in terms of reduced microleakage and increased bond strength [10].

Micrographs of Er:YAG laser irradiated dentin exhibit no signs of carbonization or melting, but opened dentinal tubules and roughness caused partly by the ablation of more inter-tubular dentin than peri-tubular dentin [11]. Er:YAG laser creates an irregular and micro-retentive morphological pattern without hard tissue damage. Even if for dentin preparation the reviewed literature shows that Fluencies reaching 45J/cm^2 combined with pulses ranging from 200 to 400 µsec. and an output power ranging from 300mJ to 500 mJ with a 6 to 10 Hz repetition rate, result in a bond strength similar to bur-treated surfaces, the findings of the literature are still controversial regarding shear bond strength [12]. After laser irradiation, a superficial layer is formed (around 5 µm thickness) where no collagen fibrils can be identified. It is clear today that, in order to achieve high bond strength values on dentin and enamel, acid treatment of the lased dentin surfaces is able to establish a hybrid layer and tag formation [13]. Studies regarding the marginal seal of the restorations of the Er:YAG-prepared cavities indicate that acid etching is still necessary to guarantee a good result comparable to or improving the marginal seal obtained in bur-prepared cavities [14,15].

Recently, it was hypothesized that Er:YAG laser irradiation could produce chemical modifications in proteins, phosphate and carbonate contents in dentin [16].

Can these morphological and chemical changes (as compared with bur preparation) interact with adhesive systems?

Wettability, which is considered as essential to obtain good dentin bonding for adhesive system [17], depends on roughness as well as surface tension: so, measurements were taken on Er:YAG laser-irradiated dentin for comparison with conventional bur preparation.

All comparisons were made on the same dentin specimen thus avoiding the differences caused by substrate heterogeneity.

Materials and methods

Samples preparation

Eleven freshly-extracted permanent human third molars were selected and stored at 4°C in distilled water for periods of up to three months prior to the experiment. Only caries-free and restoration-free teeth were used. These samples were collected conforming to a protocol that satisfied the ethical standards as described by the "*Centre Hospitalier Universitaire de Nice*". Teeth were extracted for periodontal reasons and the patients consented orally to their use for research purposes. All the teeth were embedded in clear epoxy resin (Buehler® Ltd., Lake Bluff, IL, USA). A first transversal section of the crown was performed 4mm from the occlusal surface with a diamond blade under continuous water flow (Isomet 2000, Buehler® Ltd. USA). A second cut was made parallel to the first in order to obtain 1mm thick lamellae. The dentin surface was then divided into two equal areas. The first half was prepared using a round carbide bur (ref. H1.204.018, Komet, Gebr Brasseler, Lemgo, Germany) and a high-speed handpiece plus air-water spray. The second half was prepared using an Er:YAG laser (Key III™, Kavo, Biberach, Germany) with the following parameters: mirror handpiece, air-water spray, working distance 12mm, pulse time 400µs, output power 350mJ, repetition rate 10 Hz, spot size diameter 0.8mm, fluence 44.5J/cm^2. Laser irradiation scanned the target surfaces perpendicularly. A control of the spots confluence was performed with a microscope (original magnification x 10, Olympus MTV-3, Japan,) for all the samples. One specimen was gold coated (ion sputter Jeol JFC-1100-E, Tokyo, Japan), underwent an SEM observation and served to determine the ablation feature **(Figure 1)**.

Surfaces Roughness

Vertical scanning interferometry (VSI, Wyko NT 1100, Veeco Instruments, Inc.,Woodbury, New York) provides a way to measure the depths of rough surfaces. The light reflected from the sample reference surface recombines in constructive and destructive interference patterns (so-called fringes), producing a characteristic map. The VSI evaluates this map (pattern of fringes) to provide highly accurate information about surface characteristics of the tested samples. Parameters measured using this technique are the same as those obtained using a mechanical profilometry (Ra and Rt). Ra is the mean roughness of the analyzed area and Rt denotes the distance between the highest peak and the

Figure 1: Spots confluence: The upper half was prepared using a round carbide bur (ref. H1.204.018, Komet, Gebr Brasseler, Lemgo, Germany) and a high-speed handpiece plus air-water spray while the lower was prepared using an Er:YAG laser (Key III™, Kavo, Biberach, Germany) with these f parameters: mirror handpiece, air-water spray, working distance 12mm, pulse time 400μs, output power 350mJ, repetition rate 10 Hz, spot size diameter 0.8mm, fluence 44.5J/cm^2.

lowest valley. Three-dimensional images of the surfaces are generated and the software enables calculations of various surface parameters and image processing. The software can calculate the volume of material between peaks and valleys (vertical resolution 0.1nm, lateral resolution 0.5nm). Three specimens, divided into two parts (six surfaces), were gold coated (ion sputter Jeol JFC-1100-E, Tokyo, Japan) and Ra and Rt were measured.

Wettability or Contact angle measurement

Drops of distilled water were deposited on the treated surfaces and the contact angles (θ), on the left and on the right, were measured. The contact angle is the angle between the dentin surface and the tangent of the drop **(Figure 2)**. The closer the contact angle reaches zero, the better the wettability. Contact angle measurements were taken using a Kruss G1 goniometer (Krüss GmbH, Hamburg, Germany). This system is composed of several components:

1. A closed chamber (constant temperature and pressure conditions) in which the sample can be moved in every direction using a mechanical cylinder.
2. A syringe, plus needle that contains the distilled water, fixed on a holder.
3. A lighting system including color filters to obtain a monochromatic light in order to avoid interfering observations.

The needle was used to dispense one drop of distilled water on the dentin surfaces. Left and right angles were measured and the time the water drop took to spread out was recorded. Wettability measurements were taken on 5 samples divided into two parts. The process was repeated for each surface, making a total of 20 measurements.

Surface Tension

The same methodology was performed on two dentin

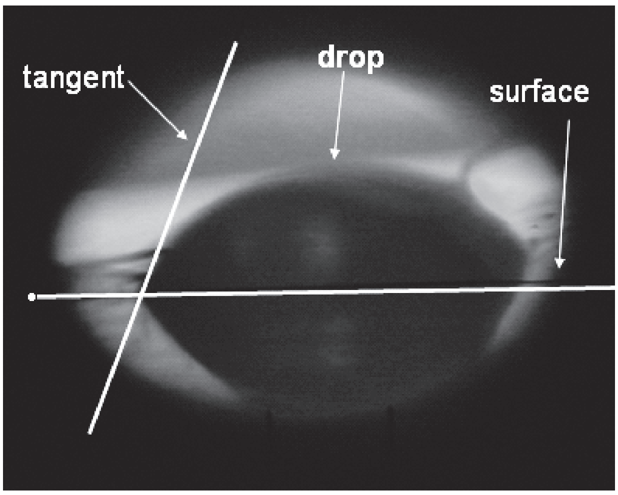

Figure 2: Water drop and Contact angle tangent measurement.

lamellae: one untreated surface (control sample) and one divided into two parts (bur *vs* laser). Observations were performed before and after gold coating (ion sputter Jeol JFC-1100-E, Tokyo, Japan), a total of twelve measurements (3 surfaces, 6 drops before and after gold coating). Contact angles were then measured as previously described.

Results

SEM observations:

SEM observation confirms that the bur-prepared dentin surface is covered with large amounts of smear layer while the Er:YAG prepared surface is smear layer free with large opened dentinal tubules. **(Figure 3)**

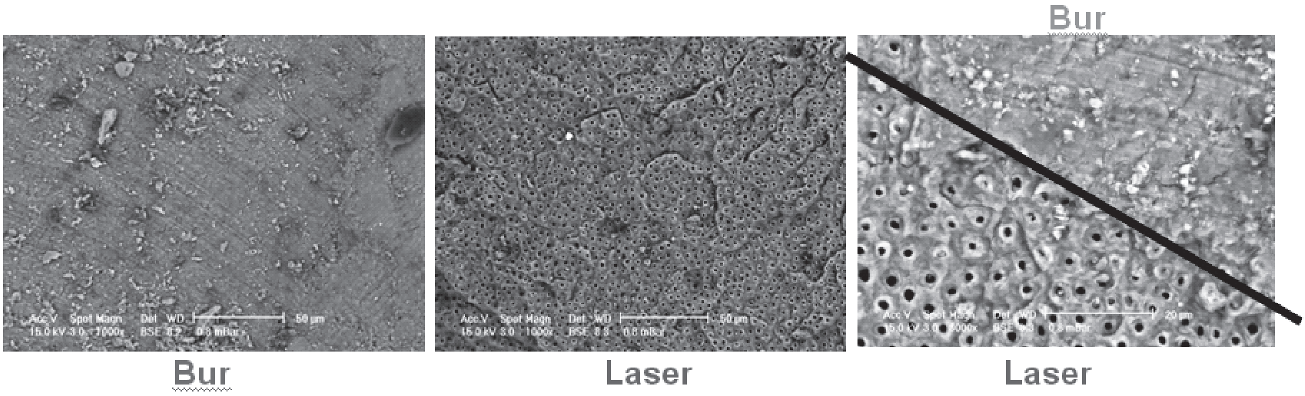

Figure 3: Observations of bur (3a), Er:YAG laser (3b) and mixed surfaces(3c) with SEM. Original magnification x1000, bar= 50µm

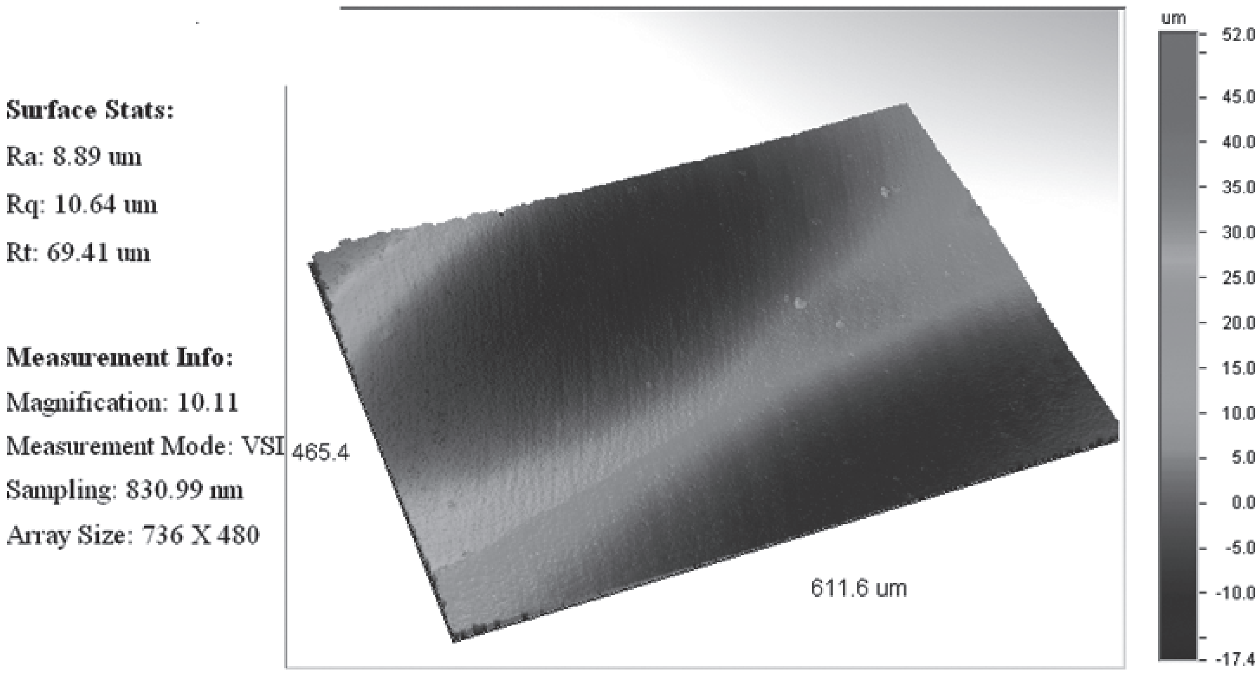

Figure 4: VSI observation of a bur-prepared dentin surface

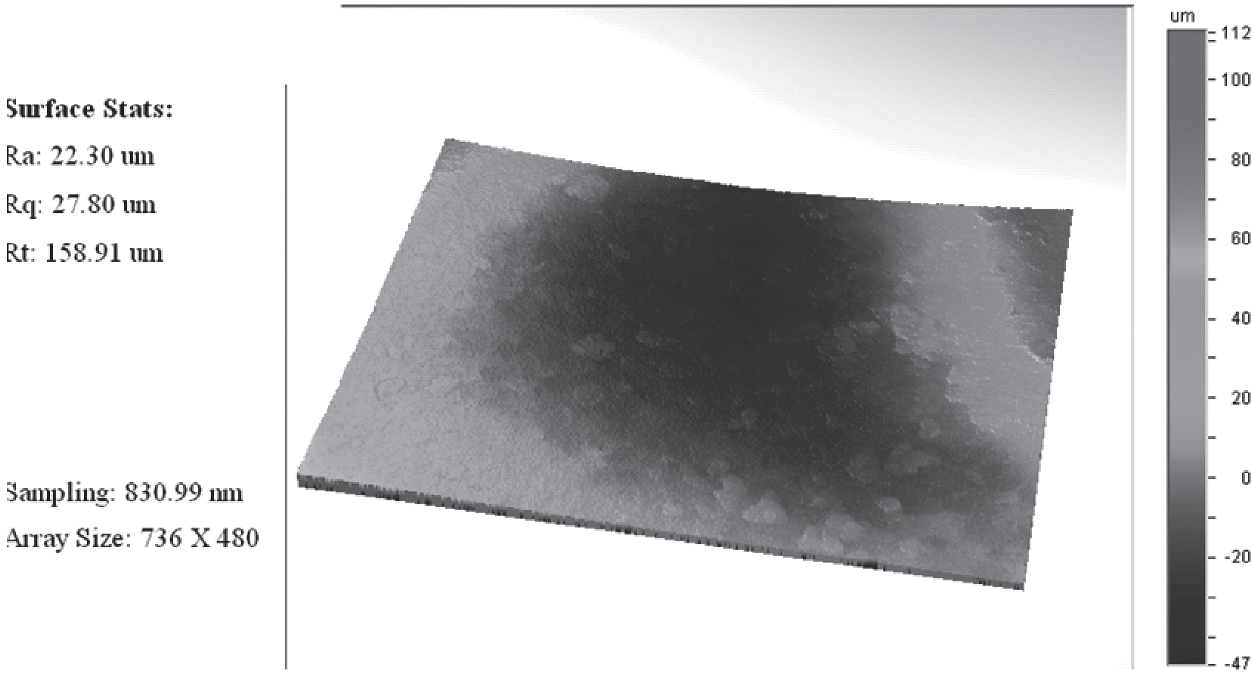

Figure 5: VSI observation of an Er:YAG laser-prepared dentin surface

Table 1: Ra and Rt measurements.

	bur		Laser	
	Ra (µm)	Rt (µm)	Ra (µm)	Rt (µm)
Sample 1	8.89	69.41	22.30	158.91
Sample 2	8.78	66.49	16.42	120.30
Sample 3	12.83	86.27	16.19	85.82

VSI observations:

Figure 4 shows the roughness profile obtained on an area of 365 x 166 µm for a bur-prepared surface, and **Figure 5** shows the roughness profile of the Er:YAG laser-prepared surface on an area of 736 x 480 µm.

Roughness profiles of bur-prepared surfaces are lower compared with Er:YAG laser-prepared surfaces, as shown in **Table 1.**

Wettability

Left and right angles were measured as well as the spreading time and mean values are presented in **Table 2**.

For the bur-prepared surfaces, the left and right angles are consistently almost equal. The average time for complete spreading ranged from 10sec. to 48sec.

For the laser-prepared surfaces, the left and right angles could not be measured and the time before complete spreading is very short (less than 3sec.).

Surface Tension

The objective of this experiment (wettability) was to study the influence of gold coating in order to dissociate the role of roughness from that of surface tension.

Two water drops were placed on both surfaces (untreated *vs* bur/laser-prepared). Regarding the untreated surface, the left angle was approximately equal to the right angle. The average time of complete water drop spreading was 120 sec. For the bur-prepared surfaces, the left angle was approximately equal to the right angle. The average time of complete water drop spreading was 70 sec.

The same experiment was performed on the irradiated surfaces : the left and right angles were lower or equal to 10° and the complete spreading time was very short, less than 3 sec. **(Table 3)**.

Before gold coating the results obtained from untreated surfaces and bur-prepared surfaces were very close, and similar to those measured after gold coating.

Considering that after gold-coating, the same surface tension was observed whatever the surface preparation, the objective was to check and control that roughness is the only parameter influencing the water drop spreading.

Table 2: Angles and spreading time of water drops (B = bur; L = laser)

	Bur preparation	**Left angle (°)**	**Right angle (°)**	**Spreading time (sec.)**	**Er:YAG laser preparation**	**Left angle (°)**	**Right angle (°)**	**Spreading Time (sec.)**
SAMPLES	B 1 drop 1	22	35	10	L 1 drop 1	8	10	3
	B 1 drop 2	37	29	20	L 1 drop 2	5	7	3
	B 2 drop 1	23	34	15	L 2 drop 1	0	0	0
	B 2 drop 2	25	30	20	L 2 drop 2	0	0	0
	B 3 drop 1	38	36	20	L 3 drop 1	0	0	0
	B 3 drop 2	22	25	40	L 3 drop 2	0	0	0
	B 4 drop 1	38	25	40	L 4 drop 1	0	0	0
	B 4 drop 2	38	29	45	L 4 drop 2	0	0	0
	B 5 drop 1	35	25	48	L 5 drop 1	0	0	0
	B 5 drop 2	32	29	35	L 5 drop 2	0	0	0
	Mean	31.0	29.7	29.3	Mean	1.3	1.7	0.6
	S.D.	7.2	4.1	13.7	S.D.	2.8	3.7	1.3

Table 3: Angles and spreading time of water drops before and after gold coating

	Left angle (°)	**Right angle (°)**	**Spreading time (sec.)**
Before gold coating			
untreated drop 136	39	120	
untreated drop 2	39	43	120
Bur drop 1	34	36	70
bur drop 2	35	29	54
laser drop 1	7	10	3
laser drop 2	8	10	3
After gold coating			
untreated drop 1	37	40	100
untreated drop 2	32	35	100
bur drop 1	25	29	56
bur drop 2	27	33	52
laser drop 1	40	40	100
Laser drop 2	40	35	118

Discussion

The role of an adhesive system is to impregnate the acid-etched and subsequently demineralised dentin surface in order to create a hybridization process, considering that spreading is linked to wettability that depends itself on roughness and surface tension.

Roughness is defined by measuring the difference between peaks and valleys on the prepared surfaces. As a consequence of high roughness values, an inner contact between dentin and the adhesive is observed [18]. Roughness and the scaly aspect of the Er:YAG laser-irradiated surfaces were studied and the dentin ablation rate was first evaluated by Hibst and Keller [19]. However, no comparisons of bur- *versus* Er:YAG laser-preparations on the same dentinal surfaces have been presented in terms of wettability.

Considering that wettability is defined by the ability of a liquid to spread out on a surface and in order to dissociate the role of roughness from the role of surface tension which represents the ability of extreme surface atoms to bond with the liquid, we decided to coat the bur-prepared and Er:YAG-prepared dentinal surfaces with gold (ion sputtering). Subsequently, superficial energy, in these conditions, becomes identical. Thus it became possible to compare the influence of surface roughness on water drop spreading time.

The dentinal macroscopic aspect of bur- *versus* Er:YAG laser- preparation is clearly different: flat with grooves for the bur-prepared surfaces and chalky with multiple spots for the irradiated surfaces. At microscopic magnifications, the smear layer covers the bur-prepared surfaces whereas laser-prepared surfaces are clean (without mud and debris). The irradiated surfaces possess increased surface porosity (dentinal tubule entries) and, at 10^{-2}mm roughness was different in comparison with the bur-prepared surfaces.

Recent studies have been performed on dentin surfaces, to compare self-etching primers and phosphoric acid preparations. It was demonstrated that they have similar increase in water wettability. However, the dentin roughness increase was lower with self-etching primers than with phosphoric acid and the presence of a smear layer did not affect the results [20]. Additional studies showed that water contact angles were lower on hydrated smear layers. No difference was found between contact angles on completely and briefly air-dried smear layers when a primer was used [21].

In this study, both bur- and Er:YAG-prepared dentin surfaces were gold coated to check the influence of roughness on wettability. Concerning bur-prepared surfaces, wettability values are almost identical before and after metallization. Superficial energy does not influence the results and roughness appears to be the main parameter involved in water drop spreading. Concerning irradiated surfaces, water drop spreading time is close to 110 sec. The water drop remains spherical as on untreated surfaces.

Conclusion

The specific morphology of Er:YAG laser-prepared dentin surfaces does not affect the wettability that depends on surface tension and porosity more than on surface roughness. Surface tension does not influence the results and roughness appears to be the main parameter involved in water drop spreading and this may be an indication, by the clinical point of view, about the choice of Er:YAG laser parameters in conservative dentistry.

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